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REPORT

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DESULPHURIZATION OF LOW-ALLOY STEEL IN A DIRECT
CURRENT ESR FURNACE

Richard C. Andrew and George M. Weston

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14 **MRL-R-806**

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Eleven small ingots (70-mm diameter, 500-mm long) were produced in an experimental electroslag-refining furnace using electrode negative and electrode positive direct current power modes to enable the extent of desulphurization to be compared. The electrode material was a commercial low alloy steel (En 25) containing about 0.03 wt% sulphur. The average sulphur reduction determined for each ingot was found to increase as the ingress of oxygen to the molten slag was reduced, and extensive desulphurization was achieved with both power modes provided the entry of oxygen to the slag bath was sufficiently restricted. Slightly lower levels of sulphur removal were recorded for the electrode positive power mode, but whether this was due solely to electrochemical reactions or to the asymmetric pattern of heat generation is uncertain.

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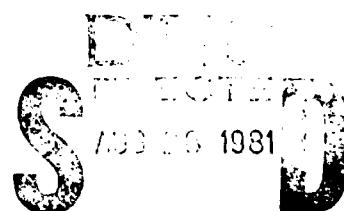
DOCUMENT CONTROL DATA SHEET

16. **ABSTRACT** (if this is security classified, the announcement of this report will be similarly classified):

Eleven small ingots (70-mm diameter, 500-mm long) were produced in an experimental electroslag-refining furnace using electrode negative and electrode positive direct current power modes to enable the extent of desulphurization to be compared. The electrode material was a commercial low alloy steel (En 25) containing about 0.03 wt% sulphur. The average sulphur reduction determined for each ingot was found to increase as the ingress of oxygen with both power modes provided the entry of oxygen to the slag bath was sufficiently restricted. Slightly lower levels of sulphur removal were recorded for the electrode positive power mode, but whether this was due solely to electrochemical reactions or to the asymmetric pattern of heat generation is uncertain.

C O N T E N T S

DESULPHURIZATION OF LOW-ALLOY STEEL IN A DIRECT

CURRENT ESR FURNACE

1. INTRODUCTION

The electroslag refining (ESR) process is known to improve ingot quality
in several ways, one of which is a reduction in inclusion content, notably
sulphides. The reactions which control the extent of desulphurization have
been discussed in detail by several authors [1-3]. Briefly, the transport of
sulphur from metal to slag occurs by the reaction

$$[S] + (O^=) \rightleftarrows (S^=) + [O] \qquad \qquad ...(1)$$
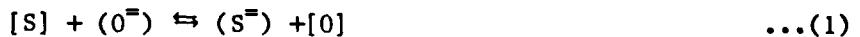
Where [ ] and ( ) refer to metal and slag respectively. Assuming Henrian
behaviour [1], the partition coefficient (L) for this reaction may be written:

$$L = \frac{wt\% (S^=)}{wt\% [S]} = a \text{ constant} \times \frac{N_{(O^=)}}{N_{[O]}} \qquad ...(2)$$

where $N_{(O^{2-})}$ and $N_{[O]}$ refer to the mole fraction of O^{2-} ions in the slag and O
atoms in the metal respectively. Thus, desulphurization is promoted by high
slag basicity and a low concentration of oxygen in the metal bath.

In the electroslag refining of ferrous alloys, the oxygen content of the
metal is related to the activity of FeO in the slag by the reaction

$$(FeO) \rightleftarrows [Fe] + [O] \qquad \qquad ...(3)$$

Therefore, to achieve low oxygen potentials in both the slag and the metal so
as to facilitate sulphur removal, the FeO activity of the slag bath must be
kept low. Typically in ESR practice, the FeO level in the slag is no more
than 0.3-0.6 wt% to ensure effective sulphur removal. Provided this level is

not exceeded, the oxygen activity in the metal is so low that the sulphur capacity of the slag is determined simply by slag basicity [4,5]. If the sulphur capacity of the slag is low, or if the amount of sulphur to be removed is large, it becomes necessary for sulphur to be transferred from the slag to the furnace atmosphere; otherwise the metal-slag sulphur transfer reaction will effectively cease. This transfer of sulphur occurs by the following reaction:

$$(S^-) + 3/2 \{O_2\} \leftrightarrows \{SO_2\} + (O^-) \qquad \ldots(4)$$
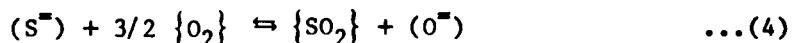
where $\{\ \}$ refers to the gas phase. The distribution coefficient for this reaction is given by:

$$L = a \text{ constant} \times \frac{P^{3/2}_{O_2}}{N_{(O^{2-})}} \qquad \ldots(5)$$

where L is the distribution coefficient of sulphur between gas and slag and where $P_{\{O_2\}}$, $N_{(O^{2-})}$ are the partial pressure of oxygen in the gas phase and the mole fraction of O^{2-} ions in the slag, respectively. A high oxygen partial pressure in the furnace atmosphere aids this stage of the desulphurization process along with a low slag basicity. For a wide range of slag compositions an air atmosphere in the mould cavity provides sufficient oxygen for reaction (4) to proceed to the right throughout a long term remelting operation.

Evidence is now well documented that reactions (1) and (4) function to enable sulphur removal of around 70-90% to be obtained routinely when ESR furnaces are operated in an air environment with adequate deoxidation, basic slags, and alternating current power supplies [6,7].

However, the position is more uncertain when direct current ESR furnaces are operated under similar conditions, possibly because of the additional complication of electrochemical reactions [1]. Much of the data which has been published on desulphurization in d.c. furnaces is summarised in Table I. The extent of the disagreement about the relative desulphurizing abilities of the two d.c. modes is clearly evident from this Table. While most authors claim that sulphur removal is better with the d.c. electrode positive mode, particularly in small ingot sizes, others report good sulphur removal in larger ingots remelted in commercial furnaces operated with either d.c. positive [13,14] or d.c. negative [12] power modes. The results presented by Holzgruber et al [11] show that, for the electrode positive polarity using small diameter ingots, good desulphurization could only be achieved for ingots below 150 mm diameter. The effect was reversed for the opposite polarity. The authors attribute this sudden change in behaviour to the important role that current density plays in electrochemical reactions.

It is also obvious from Table I that essential remelting details for both the experimental and the commercial ingots have not been supplied by most authors. In particular, information is lacking on deoxidation practice, specific slag compositions, and furnace atmospheres. As these factors

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strongly influence the thermochemical reactions for sulphur removal, it is difficult to draw meaningful conclusions solely from the information presented in Table I.

Accordingly, ingots have been produced at MRL to enable the extent of desulphurization in each d.c. mode to be compared. Particular attention was given to isolating the processing variables which affect thermochemical reactions so that the full effect of electrochemical reactions on sulphur removal could be observed. The results of this work are contained in this report.

2. EXPERIMENTAL

Eleven ingots with a nominal diameter of 70 mm and with lengths up to 500 mm were produced in an experimental ESR furnace which has been described in detail elsewhere [16]. The remelting conditions for each of these ingots are summarised in Tables II and III, and are separated below into four categories on the basis of the procedures used to vary oxygen access to the molten slag bath:

    (i)   unrestricted oxygen access - electrodes with their surface scale intact; remelted in an air atmosphere (ingots 7 and 32)

    (ii)   partial oxygen restriction - electrodes with their surface scale removed by grinding; remelted in an air atmosphere (ingots 29 and 34)

    (iii)   oxygen ingress severely restricted - descaled electrodes, remelted in an argon atmosphere (ingots 38, 39 and 40)

    (iv)   oxygen ingress severely restricted - descaled electrodes, remelted with a portion of the slag charge maintained in the unfused granulated condition above the liquid slag pool (ingots 10, 12, 36 and 41).

The electrode material was commercial En 25 steel containing approximately 0.03 wt% sulphur. Deoxidants were not added to the slag bath during the remelting of any of these ingots. Two slag compositions were used to simulate commercial production which generally is based on 2 and 3 component slags (Table II).

An inert atmosphere was achieved by first purging the mould cavity with argon for several minutes prior to start-up and then maintaining this flow throughout the remelting operation. The upper mould orifice was almost completely sealed to further restrict the ingress of oxygen. An unfused slag cover above the slag bath was achieved by adding powdered slag to the mould each time liquid slag broke through to the surface. While few such additions were needed when operating with the electrode negative mode, the creation and maintenance of a physical barrier to oxygen movement in this manner was much more difficult with the electrode positive mode. Melt rates were also

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generally higher for the electrode positive mode (Table II) and the remelting conditions more unstable. For example the production of ingot 39 was terminated prematurely because of erratic remelting conditions. An ingot subsequently was produced successfully under similar experimental conditions (ingot 40).

Remelting conditions were deliberately varied during the production of two of the ingots. In the first case (ingot 12), the bulk of the electrode length was remelted under an unfused slag cover but when three-quarters of the desired ingot length had been obtained molten slag was allowed to breach and subsequently consume this cover. In the second case (ingot 29), the ingot initially was formed with the electrode deeply immersed in the slag bath which supported a light cover of unfused slag; the electrode was then withdrawn until its tip was just immersed in the slag bath and the final one-third of the ingot length was formed with the electrode in this position.

All ingots were sectioned longitudinally and analysed for sulphur at 12.5 mm intervals along the ingot centre-line for the first 100 mm of ingot formation and thereafter at 50 mm intervals. Samples were also taken from some ingots for oxygen determination by a vacuum fusion technique in which the gases evolved were analysed by mass spectrometry. Wet chemical methods were used to determine the FeO content of each slag cap.

3. RESULTS

Oxygen levels were determined at selected positions in a sufficient number of ingots to cover almost the full range of experimental conditions outlined in the previous section. The results are given in Table III along with the sulphur contents of each electrode, the average percentage sulphur reduction* ($S_{elect.} - S_{ingot}/S_{elect.} \times 100$) obtained when it was remelted, and the FeO content of the corresponding slag cap at the completion of remelting. Three sulphur levels are reported for each ingot in Table III to indicate the variation in degree of desulphurization along an ingot length. Full details of the influence of the different remelting conditions on desulphurization are presented in Figures 1-3.

For the electrode negative power mode, sulphur removal was high in the initial stages of ingot formation when an electrode with its surface scale intact was remelted in an air atmosphere, but then rapidly diminished (ingot 32 - Fig. 1). Resulphurization eventually occurred, raising the ingot sulphur level well above that of the electrode material. As a result, the average sulphur reduction over the entire ingot length was negligible (Table III). The oxygen content of the top of this ingot was much higher than that

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*   Calculated as the arithmetic mean of the sulphur determination along the length of each ingot.

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of the electrode; this was accompanied by a high FeO level in the slag cap. Substantial sulphur removal was obtained for a considerable portion of the length of ingot 29 which was initially remelted in an air atmosphere with the electrode tip deeply immersed in the slag bath. Desulphurization decreased suddenly when the tip was raised to the surface of the bath, the final ingot sulphur level approaching that of the electrode. Similarly uniform desulphurization was achieved beneath an unfused slag cover so long as molten slag did not break through to the atmosphere; when this happened, sulphur removal decreased (ingot 12 – Fig. 1 and Table III). Two examples of marked uniform desulphurization obtained when oxygen ingress to the slag bath was restricted until the completion of the remelting operation are given in Fig. 2. Note that desulphurization in an argon atmosphere was not as uniform or as extensive as that under an unfused slag cover.

Remelting with the electrode positive power mode also produced a pattern of sulphur removal which ranged from negligible when oxygen ingress was unrestricted (ingot 7) to substantial when either an argon atmosphere or an unfused slag cover was employed (ingots 36, 39 and 40 – Fig. 3 and Table III). The desulphurization profile for ingot 34 (descaled electrode) was similar to that for ingot 7 (as–received electrode surface) except that the initially high rate of sulphur removal was maintained for a longer period. One consequence of this was a small value for the average reduction in sulphur content (Table III).

Generally, the extent of desulphurization obtained with the electrode negative mode was slightly higher and more uniform than that observed with the electrode positive mode for comparable remelting conditions. Extensive sulphur removal in both modes was accompanied by low ingot oxygen levels and, in the electrode negative mode, by low FeO levels. By contrast, remelting with the electrode positive mode consistently produced slag caps rich in FeO. For both modes sulphur removal was comparable but not as extensive in those ingots remelted with the less basic 33% CaF_2 + 33% CaO + 33% Al_2O_3 slag.

4. DISCUSSION

The present work has established that desulphurization in direct–current electroslag remelting furnaces is largely independent of electrode polarity but is primarily governed by the amount of oxygen available to the slag bath. It should also apply to all steels providing oxygen potentials can be maintained at low levels. The mechanisms for sulphur removal in d.c. furnaces therefore closely parallel those in a.c. furnaces. Extensive desulphurization persisted for only a short time when oxygen was freely available to the slag bath, the desulphurization profiles being very similar for both d.c. modes. The desulphurization interval was lengthened by removing the surface scale from the consumable electrode indicating that, for small diameter electrodes and therefore a high surface area–to–volume ratio, any scale formed contributes a significant quantity of oxygen to the slag bath. This finding is in agreement with that reported by Latash and Medover [17]. Thus, although the transfer of sulphur from slag to furnace

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atmosphere is promoted by an oxygen-rich environment (reaction 4 - section 1), the oxygen potential of the slag bath also increases rapidly unless adequate deoxidation procedures are maintained. This increase quickly stifles the transfer of sulphur from metal to slag and desulphurization ceases (reaction 1).

Levels of sulphur removal of around 80%, which approximate those achieved in a.c. ESR furnaces, were recorded for both d.c. modes by effectively excluding oxygen from the slag bath. In this regard, the argon atmosphere did not perform as well as the unfused slag cover, probably because the arrangement used to replace the normal furnace atmosphere with argon was not able to completely exclude oxygen from the system. Accordingly, more uniform desulphurization could be expected in a fully enclosed ESR system, such a converted VAR furnace of the kind described by Grunbaum and Gufstafsson [12], which may be evacuated and then back-filled with an inert gas. The extensive desulphurization obtained in this work was accompanied by very low metal oxygen levels, similar to those of the vacuum-degassed electrode material. This result, in conjunction with the observation that sulphur removal was severely curtailed when the metal oxygen level approached 0.01 wt%, provides further evidence of the important link between the oxygen content of the slag bath and sulphur removal in d.c. furnaces. Grunbaum and Gufstafsson [12] have also reported only minor desulphurization in d.c. furnaces when the metal oxygen content reached the above value.

The dominant role of thermochemical reactions in sulphur removal in d.c. furnaces was also emphasized by the results of the two trials in which stable remelting conditions were deliberately perturbed near the end of ingot formation. In both instances, the main consequence of the change in conditions was to increase oxygen availability to the remelting zone, the electrical parameters being unaltered apart from an increase in voltage when the electrode immersion depth was varied. For example, direct oxygen transfer from the furnace atmosphere to slag bath (via the molten slag bath surface) became possible by deliberately allowing molten slag to breach the powdered slag cover (ingot 12). Raising the electrode tip from deep within the slag bath to the slag bath surface also increased the role of electrode surface oxidation as a source of oxygen to the slag bath (ingot 29). Latash and Medover [17] have observed that, with a shallow electrode immersion depth, the temperature of a short segment of electrode immediately above the slag bath surface may reach 1200°C, leading to extensive surface oxidation. This oxide may produce a significant increase in the oxygen content of the remelted ingot, possibly by dissolution of oxide directly into the liquid metal droplets. Latash and Medovar [17] also reported that deep electrode immersion substantially reduced oxidation of the electrode surface. In the present trials, the perturbations in melting conditions resulted immediately in a rapid rise in the ingot sulphur content.

The slightly higher level of desulphurization determined for the electrode negative power mode suggests that electrochemical reactions operate in d.c. furnaces, promoting sulphur removal more at one electrode polarity than the other. On the basis of the most frequently cited electrochemical

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reaction for desulphurization [18]

$$[S] + 2e \leftrightarrows (S^=) \qquad \qquad ...(6)$$

the negative pole is the optimum location for the transfer of sulphur from metal to slag. Mellberg and Sandberg [15] proposed that this reaction is enhanced when the electrode is negative because of the higher current density and therefore the greater potential drop across the electrode/slag interface. While the results of the present work appear to support this proposal, other factors, notably asymmetrical heat generation, may also have caused the difference in degree of sulphur removal between the two modes.

With the electrode negative, the majority of the heat was generated at the metal pool/slag interface. An unfused slag cover was easily maintained with this configuration indicating that, for the slag volumes and electrode immersion depths used in the present work, the upper portion of the liquid slag bath was comparatively cool. When the electrode was made the positive pole, heat generation was concentrated at the electrode/slag interface, i.e. much closer to the upper surface of the slag bath for similar slag volumes and electrode immersion depths to those used with the other polarity. Consequently, the unfused slag cover was frequently breached by molten slag. The asymmetric heat generation also produced greater oxidation of the electrode surface at the slag bath/furnace atmosphere interface when the electrode was positive. Thus, oxygen access to the slag bath would have been more difficult to control when remelting with positive polarity. This may explain the observed lower and more variable degree of sulphur removal, and the higher slag FeO contents.

5. CONCLUSIONS

1. Extensive desulphurization can be achieved using either electrode positive or electrode negative direct current power modes so long as the access of oxygen to the slag bath is restricted. Thus, sulphur removal in small direct current ESR furnaces is predominantly controlled by thermochemical reactions.

2. Electrochemical reactions may have been responsible for the slightly lower level of desulphurization recorded for the electrode positive mode. However, the asymmetric pattern of heat generation made the operating conditions less stable in this mode and may therefore have substantially influenced the degree of sulphur removal.

3. Surface scale on the wrought electrodes as well as oxide formed immediately above the slag bath surface during the remelting operation contribute significant amounts of oxygen to the slag bath. The degree of ingot desulphurization is reduced as a consequence.

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TABLE I

Summary of results reported for the influence of power mode on extent of desulphurization in the ESR Process

AUTHORS	STEEL TYPE	INGOT SIZE	NOMINAL SLAG COMPOSITION	FURNACE ATMOSPHERE	DEOXIDATION PRACTICE	DEGREE OF DESULPHURIZATION		
						D.C. -	D.C. +	**A.C.
Hlineny and Buzek [8]	?	75mm dia.	?	?	?	Minor	Moderate	Moderate
Whittaker et al [9]	?	90mm dia.	?	?	?	Minor	Moderate	-
*Swinden [10]	high sulphur En 1A	110mm dia.	CaF_2 + 5,10,20 and 30% CaO	Air	None	D.C. -	D.C. +	A.C.
Holzgruber et al[11]	?	75mm dia.	CaF_2 + CaO + Al_2O_3 type	?	?	Minor	Extensive	Extensive
	?	200,325 and 400mm dia.	CaF_2 + CaO + Al_2O_3 type	?	?	Extensive	Minor	Extensive
Grünbaum and Gustafsson	18/8 stainless	170mm dia.	60% CaF_2 + 40% CaO	Argon	?	Extensive	Moderate	-
	18/8 stainless and ball-bearing	?	60% CaF_2 + 40% CaO	Air	?	non-moderate	?	-

*   Desulphurization only effective for very short distance along ingot length for each power mode - the area under each ingot sulphur content versus ingot length curve used to determine relative desulphurization ratings.

** Comparison with A.C. power mode is included because nearly all commercial ESR steel is produced this way, and is considered to be the most efficient in removing sulphur.

TABLE I
(Continued)

AUTHORS	STEEL TYPE	INGOT SIZE	NOMINAL SLAG COMPOSITION	FURNACE ATMOSPHERE	DEOXIDATION PRACTICE	DEGREE OF DESULPHURIZATION		
						D.C. –	D.C. +	**A.C.
Grunbaum and Gustafsson [12]	18/8 stainless	170mm dia. 415mm square	50% CaF_2 + 20% CaO + 30% Al_2O_3	Argon	?	non-moderate	?	–
Dewsnap and Schlatter [14]	High speed tool	300mm dia.	CaF_2 + CaO + Al_2O_3	?	?	none	Extensive	Extensive
Schlatter [14]	Low and high alloy	up to 600mm dia.	CaF_2 + CaO + Al_2O_3 and CaF_2 + Al_2O_3 + SiO_2 types	Air	?	none	Moderate	Extensive
Mellberg and Sandberg [15]	high sulphur high speed tool	100mm dia.	70% CaF_2 + 15% CaO + 15% Al_2O_3	Air	?	Minor	Extensive	Extensive
Andrew and Weston (This work)	Low alloy	70mm dia.	Details are summarised in Table III			Extensive	Extensive	

** Comparison with A.C. power mode is included because nearly all commercial ESR steel is produced this way, and is considered to be the most efficient in removing sulphur.

TABLE II

Summary of remelting conditions

No.	Ingot		Polarity	Electrode			Slag		Current A	Voltage V
	Dia. mm	Length mm		Dia. mm	Feed Rate m/h	Melt Rate Kg/h	Nominal Compn. $(CaF_2-CaO-Al_2O_3)$, wt%	Mass, g		
10	70	500	- ve	50	0.3	17.9	70-30-0	1200	800	20
12	70	400	- ve	50	0.25	15.6	70-30-0	1200	900	19
29	70	350	- ve	45	0.65	30.1	33-33-33	900	780-800	25-35
32	70	430	- ve	40	0.80	30.6	70-30-0	900	800-850	28
38	70	350	- ve	50	0.40	25.3	70-30-0	900	900-950	23
41	70	175	- ve	40	1.05	43.7	33-33-33	1050	800-900	20
7	70	455	+ ve	50	0.50	27.0	70-30-0	1200	700	18-24
34	70	460	+ ve	45	1.00	46.8	70-30-0	800	850	20
36	70	355	+ ve	45	0.80	45.0	70-30-0	1300	850	15-23
39	70	140	+ ve	50	0.60	41.2	33-33-33	900	800-900	22-25
40	70	430	+ ve	40	1.30	51.4	33-33-33	1300	900	20

TABLE III

Experimental Results

Ingot Number	Electrode Polarity	Oxygen Restriction[©]			FeO in Slag Wt%	Oxygen, Wt%			Sulphur, Wt%				Average Sulphur Reduction %
		Unfused Slag Cover	Argon Cover	None		Elect.	Ingot Base	Ingot Top	Elect.	Ingot Base	Ingot Mid-Length	Ingot Top	
10	– ve	X			0.44	0.001	0.0005	0.001	0.031	0.007	0.004	0.006	84
12[+++]	– ve	X			0.09	0.0009	0.0006	0.007	0.030	0.002	0.004	0.015	88/67
29[++]	– ve			X	0.26	0.001	0.007	0.006	0.026	0.012	0.012	0.020	53/21
32*	– ve			X	0.80	0.001	0.006	0.012	0.025	0.004	0.037	0.024	5
38	– ve		X		0.09				0.030	0.003	0.01	0.01	75
41	– ve	X			0.23				0.026	0.01	0.01	0.01	60
7*	+ ve			X	0.86				0.030	0.012	0.033	0.036	0
34+	+ ve			X	0.65	0.001	0.014	0.020	0.025	0.013	0.028	0.019	13
36	+ ve	X			0.76	0.001	0.0006	0.003	0.026	0.006	0.007	0.007	73
39**	+ ve		X						0.030	0.012	0.009	0.012	63
40	+ ve		X		0.98	0.001	0.005	0.007	0.027	0.015	0.012	0.013	48

©   Method used to restrict oxygen transfer from the furnace atmosphere to the slag bath.

*   Electrode surface not descaled prior to remelting.

**   Short ingot length.

+   Wide variation in sulphur content along ingot length.

++   Average sulphur reduction measured prior to change in electrode immersion depth/after change in immersion depth.

+++   Average sulphur reduction measured prior to, and after, the unfused slag cover was breached by molten slag.
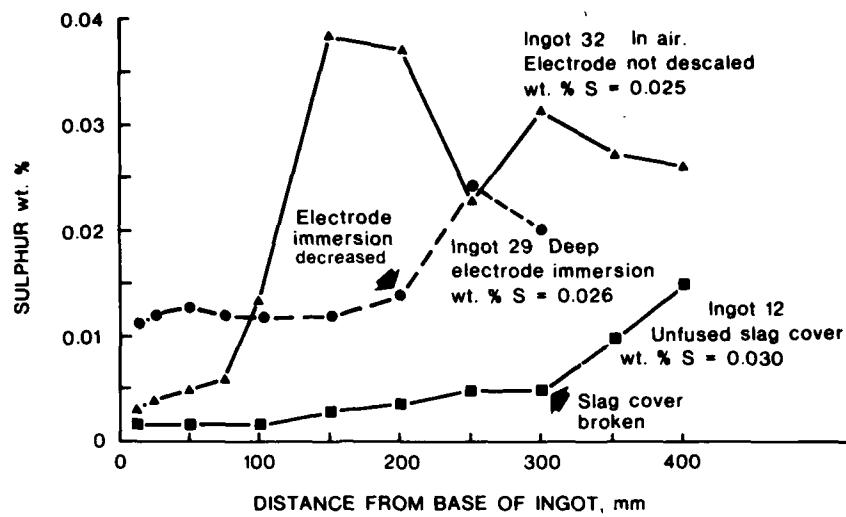
FIG. 1 – Influence of remelting conditions on the extent of
desulphurization achieved with the electrode negative
power mode. The sulphur contents of the parent
electrode are also shown.
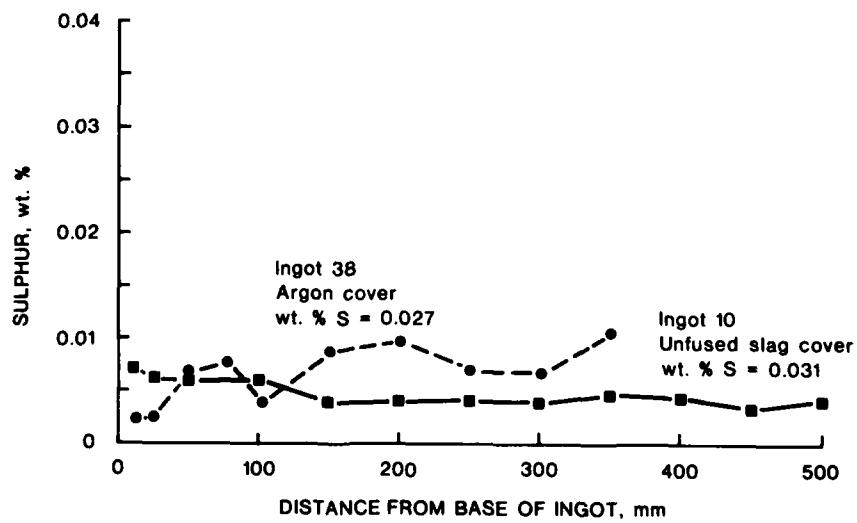
FIG. 2 – Comparison of the extent of desulphurization achieved
with argon cover and an unfused slag cover – electrode
negative power mode. The sulphur contents of the
parent electrodes are also shown.
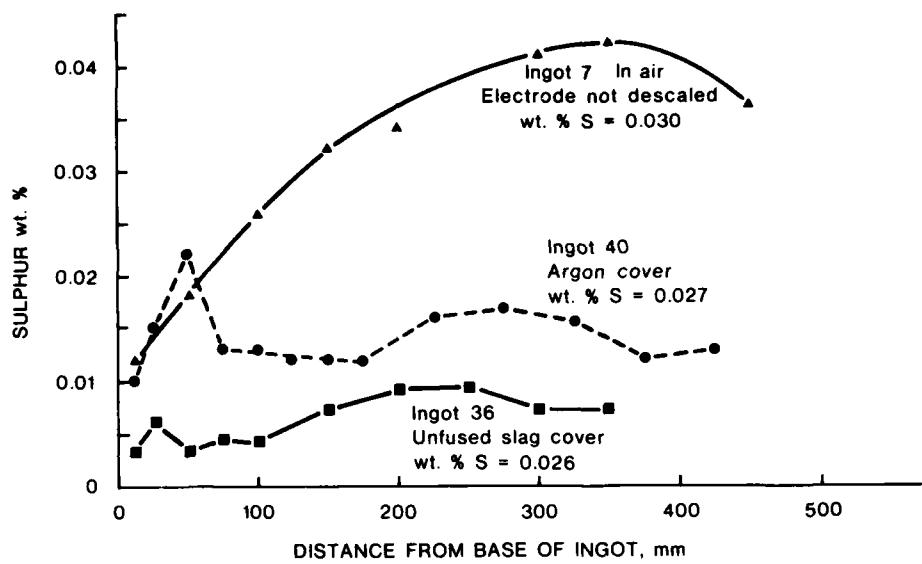
FIG. 3 – Influence of remelting conditions on the extent of
desulphurization achieved with the electrode positive
power mode. The sulphur content of each parent
electrode is also shown.

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